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Supporting Information

Efficient Stereochemical Relay En Route to Leucascandrolide A

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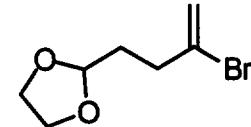
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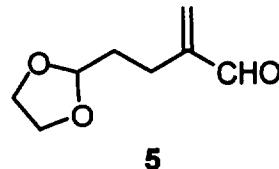
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ether (200 mL) and water (200 mL). The organic layer was separated, washed with water (300 mL), saturated aqueous solution of NaHCO₃ (200 mL), dried over anhydrous MgSO₄. Concentration under reduced pressure gave 12.4 g of the crude aldehyde **11** (74% yield), which was used for the next step without further purification.

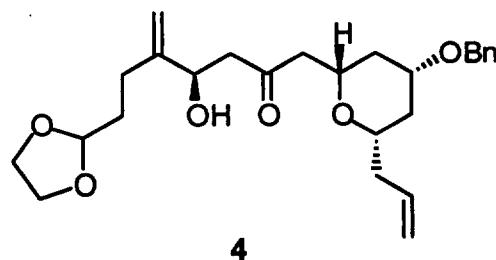
A 100-mL round-bottomed flask, equipped with the Dean-Stark trap was charged with aldehyde **11** (4.66 g, 28.6 mmol), ethylene glycol (1.86 g, 30 mmol), TsOH (50 mg) and benzene (50 mL). The resulting mixture was heated under a gentle reflux for 5 h, partitioned between ether (30 mL) and saturated aqueous solution of NaHCO₃ (50 mL). The aqueous layer was extracted with ether (30 mL), dried over anhydrous MgSO₄, filtered and concentrated. The resulting dark-brown oil was subjected to the bulb-to-bulb distillation (100 °C oven temperature, 2 mm Hg) to give 5.62 g (96% yield) of the corresponding acetal, as a pale-yellow oil. ¹H NMR (400 MHz, CDCl₃) δ 1.91 (m, 2H), 2.54 (m, 2H), 3.84 (m, 2H), 3.95 (m, 2H), 4.88 (t, 1H, J = 4.5 Hz), 5.38 (d, 1H, J = 1.7 Hz), 5.59 (q, 1H, J = 1.2 Hz); ¹³C NMR (100 MHz, CDCl₃) δ 32.3, 35.8, 65.0, 103.1, 116.8, 133.6; IR (neat) 2957, 2881, 1629, 1406, 1138, 1037, 886 cm⁻¹.



Aldehyde 5. A solution of vinyl bromide (3.70 g, 17.8 mmol), prepared in the previous step, in THF (50 mL) was treated dropwise with *n*-butyllithium (7.2 mL, 2.5 M in hexane) at -78 °C, followed by addition of dimethylformamide (6.9 mL) after a 20 min period. The reaction mixture was allowed to warm to ambient temperature, quenched by addition of water (200 mL), extracted with ether (2x300 mL), dried over anhydrous MgSO₄, filtered and concentrated. Flash chromatography on silica gel (elution with ether:hexane; 1:3, 1:1) afforded 0.81 g of starting bromide and 1.15 g of aldehyde **5** (53% yield) as a colorless oil. ¹H NMR (500 MHz, CDCl₃) δ 1.80 (m, 2H), 2.36 (t, 2H, J = 8.0 Hz), 3.83 (m, 2H), 3.94 (m, 2H), 4.86 (t, 1H, J = 4.5 Hz), 6.00 (s, 1H), 6.26 (s, 1H), 9.52 (s, 1H); ¹³C NMR (125 MHz, CDCl₃) δ 22.3, 31.7, 64.9, 103.7, 134.0, 149.5, 194.4; IR (neat) 2952, 2884, 1688, 1138, 1035, 945 cm⁻¹.

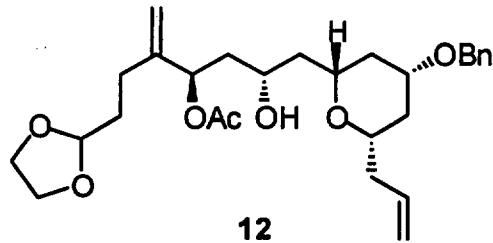
**5**

Ketone 4 Dicyclohexylboron chloride (2.5 mL of 1.0 M solution in hexane, 2.5 mmol) in ether (20 mL) was cooled to 0 °C, and treated with triethyl amine (0.39 mL, 2.8 mL), followed by addition of ketone **6** (473 mg, 1.64 mmol) in ether (6 mL). The resulting white suspension was stirred for 15 min at 0 °C, cooled to -78 °C, and treated with aldehyde **5** (442 mg, 2.83

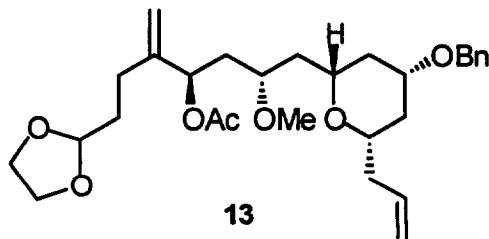
**4**

mmol) in ether (6 mL) over a 10 min period. The reaction mixture was stirred for 5 h at -78 °C, quenched by addition of 21 mL of methanol:pH 7 buffer (6:1). Upon warming to ambient temperature, 9 mL of methanol-30% hydrogen peroxide (2:1) was added. The stirring was continued for 4 h. The resulting colorless solution was partitioned between ethyl acetate (100 mL) and saturated aqueous solution of NaHCO₃ (50 mL). The organic layer was washed with 10% solution of Na₂SO₃, dried over anhydrous MgSO₄, filtered and concentrated. Flash chromatography on silica gel (elution with ethyl acetate:hexane; 1:1) afforded 554 mg of ketone **4** (76% yield) as a colorless oil. ¹H NMR (500 MHz, CDCl₃) δ 1.20 (m, 3H), 1.84 (m, 2H), 2.05 (m, 2H), 2.06-2.30 (m, 4H), 2.43 (dd, 1H, *J* = 15.0, 4.5 Hz), 2.66 (dd, 1H, *J* = 17.0, 9.0 Hz), 2.75 (m, 2H), 3.33 (m, 1H), 3.55 (dd, 1H, *J* = 11.0, 11.0, 5.0, 5.0 Hz), 3.75 (m, 1H), 3.83 (, 2H), 3.94 (m, 2H), 4.52 (m, 1H), 4.53 (s, 2H), 4.88 (m, 2H), 5.01 (d, 1H, *J* = 9 Hz), 5.03 (d, 1H, *J* = 16.5 Hz), 5.08 (s, 1H), 5.75 (dd, 1H, *J* = 17.0, 10.0, 7.0, 7.0 Hz); 7.26-7.34 (m, 5H); ¹³C NMR (125 MHz, CDCl₃) δ 26.1, 32.1, 37.3, 37.8, 40.3, 49.4, 49.7, 64.9, 69.7, 70.3, 72.2, 74.2, 75.2, 104.0, 110.1, 117.1, 127.6, 128.4, 134.3, 138.4, 149.2, 209.8; IR (neat) 3451, 2916, 2849, 1710, 1352, 1069, 906, 733, 697 cm⁻¹.

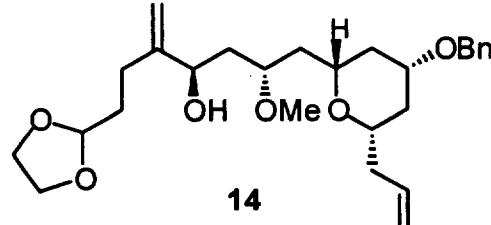
Alcohol 12. A solution of ketone **4** (120 mg, 0.27 mmol) and acetaldehyde (48 mg, 1.1 mmol) in THF (3 mL) was cooled to -10 °C and treated with SmI₂ (0.80 mL, 0.1 M solution in THF). The reaction mixture was stirred for 15 min, quenched by addition of saturated aqueous solution of NaHCO₃ (15 mL), extracted with ethyl acetate (50 mL), dried over anhydrous MgSO₄, filtered and concentrated. Flash chromatography on silica gel (elution with ethyl acetate:hexane; 2:1) afforded 122 mg of alcohol **12** (93% yield) as a colorless oil. ¹H NMR (500 MHz, C₆D₆) δ 1.15 (m, 3H), 1.60 (dt, 1H, *J* = 14.0, 9.5 Hz), 1.72 (s, 3H), 1.65-1.85 (m, 4H), 1.95 (m, 1H), 2.00-2.10 (m, 3H), 2.40 (m, 2H), 2.95 (m, 1H), 3.18 (m, 2H), 3.33 (m, 1H), 3.35 (m, 2H), 3.52 (m, 2H), 3.77 (s, 1H), 3.95 (t, 1H, *J* = 9.0 Hz), 4.34 (d, 1H, *J* = 12.5 Hz), 4.36 (d, 1H, *J* = 12.5 Hz), 4.84 (t, 1H, *J* = 4.5 Hz), 4.89 (s, 1H), 4.98 (m, 2H), 5.15 (s, 1H), 5.66 (dd, 1H, *J* = 17.0, 10.0, 7.0, 7.0 Hz); 5.87 (dd, 1H, *J* = 9.5, 2.5 Hz), 7.13 (t, *J* = 7.0 Hz), 7.22 (t, 2H, *J* = 7.0 Hz), 7.34 (d, 2H, *J* = 7 Hz); ¹³C NMR (125 MHz, C₆D₆) δ 20.7, 26.9, 32.7, 37.8, 38.8, 40.7, 42.6, 43.3, 64.8, 67.7, 69.4, 73.8, 74.3, 75.2, 76.5, 104.2, 110.4, 117.5, 127.5, 127.6, 128.6, 134.7, 139.6, 148.9, 169.6; IR (neat) 3492, 2917, 2860, 1736, 1642, 1368, 1236, 1070, 904, 736, 698 cm⁻¹.



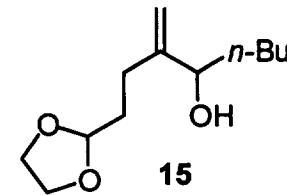
Methyl Ether 13. A solution of alcohol 12 (110 mg, 0.225 mmol), 2,6-di-*tert*-butylpyridine (0.76 mL, 3.37 mmol) and methyl triflate (0.323 mL, 2.25 mmol) was stirred for 14 h at ambient temperature, and quenched by addition of aqueous solution of ammonium hydroxide (10 mL). The product was extracted with ether (2x50 mL), dried over anhydrous MgSO₄, filtered and concentrated. The excess 2,6-di-*tert*-butylpyridine was removed by bulb-to-bulb distillation (80 °C, 0.5 mm Hg). Flash chromatography on silica gel (elution with ethyl acetate:hexane; 1:2) afforded 81 mg of methyl ether 13 (72% yield) as a colorless oil. ¹H NMR (500 MHz, C₆D₆) δ 1.19 (q, 1H, J = 11.5 Hz), 1.26 (q, 1H, J = 11.5 Hz), 1.43 (ddd, 1H, J = 14.0, 7.5, 3.5 Hz), 1.77 (s, 3H), 1.80-1.90 (m, 5H), 2.00-2.10 (m, 3H), 2.27 (m, 1H), 2.40 (m, 2H), 2.98 (m, 1H), 3.17 (s, 3H), 3.20-3.28 (m, 2H), 3.33 (m, 1H), 3.35 (m, 2H), 3.50 (m, 2H), 4.37 (s, 2H), 4.83 (t, 1H, J = 4.5 Hz), 4.89 (s, 1H), 5.03 (m, 2H), 5.17 (s, 1H), 5.84 (dd, 1H, J = 9.5, 2.5 Hz), 5.90 (m, 1H); 13C NMR (125 MHz, C₆D₆) δ 20.7, 26.8, 32.7, 38.1, 39.0, 39.4, 39.8, 40.9, 56.5, 64.8, 69.4, 72.1, 73.8, 74.7, 74.8, 75.2, 104.2, 110.5, 116.6, 127.5, 127.6, 128.5, 135.5, 139.7, 148.8, 169.3; IR (neat) 2939, 2880, 1736, 1368, 1235, 1070, 906, 736, 698 cm⁻¹.



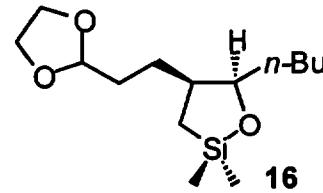
Alcohol 14. Lithium aluminum hydride (38 mg, 1 mmol) was suspended in ether (2 mL), cooled to -78 °C, and treated with methyl ether 13 (66 mg, 0.13 mmol) in ether (2 mL). The reaction mixture was stirred for 30 min at -78 °C, quenched by dropwise addition of water (0.2 mL). The resulting suspension was allowed to reach ambient temperature, vigorously stirred for 1 h, diluted with ether, dried with anhydrous Na₂SO₄, filtered and concentrated. Flash chromatography on silica gel (elution with ethyl acetate:hexane; 1:2) afforded 51 mg of alcohol 14 (86% yield) as a colorless oil. ¹H NMR (500 MHz, C₆D₆) δ 1.20 (q, 1H, J = 11.5 Hz), 1.26 (q, 1H, J = 11.5 Hz), 1.46 (ddd, 1H, J = 14.0, 7.5, 3.5 Hz), 1.80-1.85 (m, 4H), 1.93 (ddd, 1H, J = 14.0, 9.0, 4.5 Hz), 2.00-2.04 (m, 2H), 2.10 (m, 1H), 2.28 (m, 2H), 2.40 (m, 1H), 3.00 (m, 1H), 3.08 (s, 3H), 3.20-3.28 (m, 2H), 3.35 (m, 2H), 3.69 (m, 1H), 4.37 (s, 2H), 4.43 (m, 1H), 4.85 (t, 1H, J = 4.5 Hz), 4.95 (s, 1H), 5.04 (m, 2H), 5.33 (s, 1H), 5.86 (dd, 1H, J = 17.0, 10.0, 7.0, 7.0 Hz), 7.12 (t, J = 7.0 Hz), 7.22 (t, 2H, J = 7.0 Hz), 7.35 (d, 2H, J = 7 Hz); 13C NMR (125 MHz, C₆D₆) δ 26.7, 33.0, 37.9, 39.0, 38.9, 39.3, 39.5, 40.9, 56.0, 64.8, 69.4, 72.3, 74.8, 75.2, 76.4, 104.4, 108.9, 116.8, 127.5, 127.6, 128.5, 135.3, 139.7, 152.2; IR (neat) 3452, 2942, 2878, 1642, 1354, 1071, 904, 736, 698 cm⁻¹.



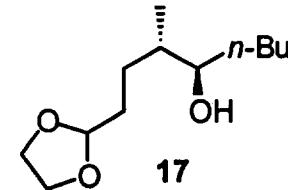
Alcohol 15. Aldehyde 5 (312 mg, 2.0 mmol) in THF (10 mL) was cooled to -78°C , and treated with *n*-butyllithium (0.88 mL, 2.5 M solution in hexane). After 15 min, the reaction was quenched by addition of saturated aqueous solution of NaHCO₃ (15 mL), extracted with ether (50 mL), dried over anhydrous MgSO₄, filtered and concentrated. Flash chromatography on silica gel (elution with ether:hexane; 2:1) afforded 380 mg of alcohol 15 (87% yield) as a colorless oil. ¹H NMR (500 MHz, CDCl₃) δ 0.87 (t, 3H, *J* = 7.0 Hz), 1.20-1.38 (m, 4H), 1.53 (m, 2H), 1.78 (br s, 1H), 1.85 (m, 2H), 2.10 (m, 1H), 2.21 (m, 1H), 3.81 (m, 2H), 3.95 (m, 2H), 4.06 (t, 1H, *J* = 7.0 Hz), 4.83 (s, 1H), 4.87 (t, 1H, *J* = 4.5 Hz), 5.01 (s, 1H); ¹³C NMR (125 MHz, CDCl₃) δ 14.0, 22.6, 25.3, 27.8, 32.2, 35.1, 64.9, 75.6, 104.2, 109.8, 151.1; IR (neat) 3439, 2953, 2870, 1692, 1409, 1135, 1031, 899, 731 cm⁻¹.



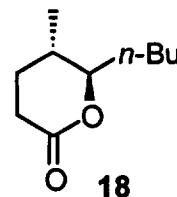
Silane 16. A solution of alcohol 15 (142 mg, 0.66 mmol) in CH₂Cl₂ (0.50 mL) was treated with tetramethyldisilazane (0.35 mL, 2.0 mmol). After 12 h, ¹H NMR analysis indicated complete consumption of the starting alcohol. The excess tetramethyldisilazane was removed under high vacuum. The resulting silyl ether was dissolved in C₆H₆ (7 mL) and treated with H₂PtCl₆ [30 μ L, 0.05 M in THF, (0.3 mol%)]. After 30 min at 50 °C, ¹H NMR analysis indicated quantitative formation of the silacycle 16, as 85:15 mixture of diastereomers. Major isomer: ¹H NMR (400 MHz, C₆D₆) δ 0.10 (s, 3H), 0.15 (s, 3H), 0.90 (t, 3H, *J* = 7.0 Hz), 1.20-1.90 (m, 10H), 2.00 (m, 1H), 3.39 (m, 2H), 3.56 (m, 2H), 3.99 (ddd, 1H, *J* = 10.4, 5.6, 3.2 Hz), 4.82 (t, 1H, *J* = 4.5 Hz); ¹³C NMR (100 MHz, C₆D₆) δ 0.2, 1.2, 14.4, 16.2, 23.0, 26.9, 28.7, 31.4, 33.2, 42.5, 64.8, 104.8; IR (neat) 2953, 2871, 1408, 1250, 1033, 840, 796 cm⁻¹.



Alcohol 17. Cyclic silane 16, obtained upon concentration of the crude hydrosilylation reaction mixture was treated with TBAF (2.6 mmol) in DMF (2.5 mL). The resulting solution was heated to 75 °C for 24 h. The excess DMF was removed by bulb-to-bulb distillation (50 °C, 0.5 mm Hg). Flash chromatography on silica gel (elution with ethyl acetate:hexane; 1:2) afforded 121 mg of alcohol 17 (82% yield from 15) as a colorless oil. ¹H NMR (500 MHz, CDCl₃) δ 0.87 (d, 3H, *J* = 7.0 Hz), 0.88 (t, 3H, *J* = 7.0 Hz), 1.20-1.90 (m, 10H), 2.74 (m, 1H), 3.41 (m, 1H), 3.82 (m, 2H), 3.95 (m, 2H), 4.83 (t, 1H, *J* = 4.5 Hz); ¹³C NMR (125 MHz, CDCl₃) δ 14.0, 15.2, 22.7, 26.0, 28.2, 31.6, 33.1, 38.6, 64.8, 75.7, 104.7.

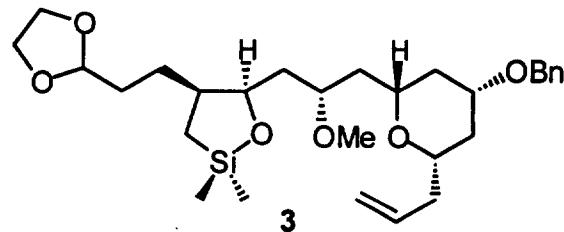


Lactone 18. A solution of alcohol **17** (110 mg, .50 mmol) in 6 mL of THF-water (5:1) containing a drop of concentrated H₂SO₄ was heated to 80 °C for 10 h, then partitioned between saturated aqueous solution of NaHCO₃ (1 mL), and ether (20 mL). The organic layer was separated, dried over anhydrous MgSO₄, filtered and concentrated. Flash chromatography on silica gel (elution with ether:pentane; 1:1) afforded 62 mg of the corresponding lactol as a colorless oil. The oxidation was conducted by addition of bromine (15 µL) to the solution of the lactol in acetic acid (0.4 mL) and water (0.64 mL). After 1 h, the reaction mixture was partitioned between saturated aqueous solution of NaHCO₃, and ether (20 mL). The organic layer was separated, dried over anhydrous MgSO₄, filtered and concentrated. Flash chromatography on silica gel (elution with ether:pentane; 1:1) afforded 61 mg of lactone **18** (86% from **17**) as a colorless oil. ¹H NMR (400 MHz, CDCl₃) δ 0.89 (t, 3H, *J* = 7.0 Hz), 0.97 (t, 3H, *J* = 7.0 Hz), 1.20-1.70 (m, 8H), 1.87 (m, 1H), 2.45 (ddd, 1H, *J* = 17.0, 10.0, 6.8 Hz), 2.60 (ddd, 1H, *J* = 17.0, 6.4, 4.4 Hz), 3.91 (ddd, 1H, *J* = 10.0, 8.0, 3.0 Hz); ¹³C NMR (100 MHz, CDCl₃) δ 13.9, 17.4, 22.6, 26.5, 27.8, 29.5, 32.2, 33.1, 85.9, 174.0.



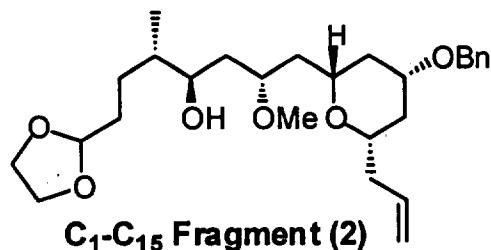
Silane 3. A solution of alcohol **13** (46 mg, 0.10 mmol) in CDCl₃ (0.50 mL) was treated with tetramethyldisilazane (0.10 mL, 0.56 mmol). After 15 min, ¹H NMR analysis indicated complete consumption of the starting alcohol. The excess tetramethyldisilazane was removed

under high vacuum. The resulting silyl ether was dissolved in C₆D₆ (1 mL) and treated with H₂PtCl₆ (0.5 mol%). After 10 min at 57 °C, ¹H NMR analysis indicated quantitative formation of the silacycle **3**, as 87:13 mixture of diastereomers. Major isomer: ¹H NMR (500 MHz, C₆D₆) δ 0.11 (s, 3H), 0.17 (s, 3H), 0.48 (dd, 1H, *J* = 14.5, 11.0 Hz), 0.80 (dd, 1H, *J* = 14.5, 7.0 Hz), 1.25 (q, 1H, *J* = 11.5 Hz), 1.36 (q, 1H, *J* = 11.5 Hz), 1.46-2.38 (m, 2H), 3.10 (m, 1H), 3.33 (s, 3H), 3.20-3.28 (m, 2H), 3.28 (m, 2H), 3.35 (m, 2H), 3.90 (m, 1H), 4.38 (s, 2H), 4.50 (m, 1H), 4.85 (t, 1H, *J* = 4.5 Hz), 5.04 (m, 2H), 5.92 (m, 1H), 7.11 (t, *J* = 7.0 Hz), 7.21 (t, 2H, *J* = 7.0 Hz), 7.34 (d, 2H, *J* = 7 Hz).

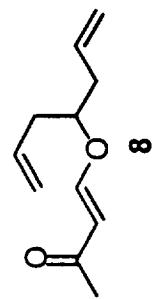


Alcohol 2. Cyclic silane **3**, obtained upon concentration of the crude hydrosilylation reaction mixture was treated with TBAF (0.4 mmol) in *d*-DMF (0.5 mL). The resulting solution was heated to 70 °C for 15 min. At this point, ¹H NMR analysis indicated complete consumption of the starting silane **3**. The excess DMF was removed by bulb-to-bulb distillation

(50 °C, 0.5 mm Hg). Flash chromatography on silica gel (elution with ethyl acetate:hexane; 1:1) afforded 24 mg of alcohol **2** (54% yield from **14**) as a colorless oil. ¹H NMR (500 MHz, C₆D₆) δ 0.93 (d, 3H, *J* = 6.5 Hz), 1.22 (q, 1H, *J* = 11.5 Hz), 1.28 (q, 1H, *J* = 11.5 Hz), 1.48-2.03 (m, H), 2.10 (m, 1H), 2.30 (m, 1H), 2.80 (br s, 1H), 3.00 (m, 1H), 3.07 (s, 3H), 3.26 (m, 2H), 3.36 (m, 2H), 3.56 (m, 2H), 3.67 (m, 1H), 3.78 (m, 1H), 4.37 (s, 2H), 4.85 (t, 1H, *J* = 4.5 Hz), 5.04 (m, 2H), 5.87 (dded, 1H, *J* = 17.0, 10.0, 7.0, 7.0 Hz), 7.12 (t, *J* = 7.0 Hz), 7.22 (t, 2H, *J* = 7.0 Hz), 7.35 (d, 2H, *J* = 7 Hz); ¹³C NMR (125 MHz, C₆D₆) δ 15.4, 27.0, 32.2, 36.3, 37.9, 38.9, 39.3, 39.4, 40.9, 56.2, 64.8, 69.4, 72.4, 74.9, 75.2, 76.8, 105.2, 116.8, 127.5, 127.6, 128.5, 135.2, 139.7; IR (neat) 3470, 2919, 2870, 1354, 1071, 736, 697 cm⁻¹.



¹H NMR and ¹³C NMR Spectra



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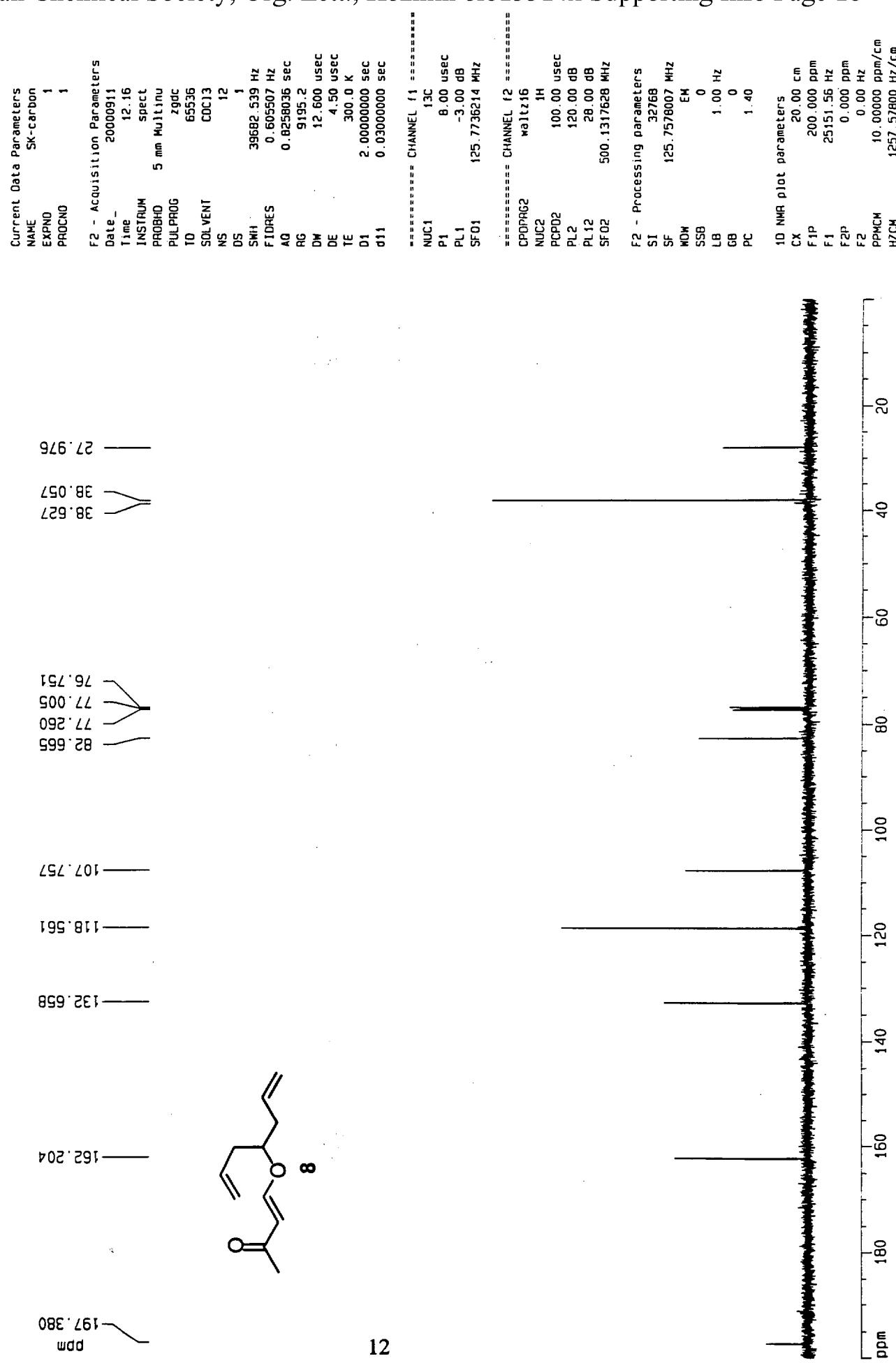
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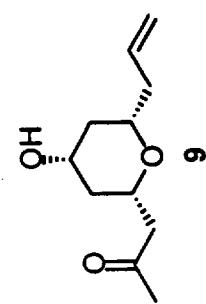
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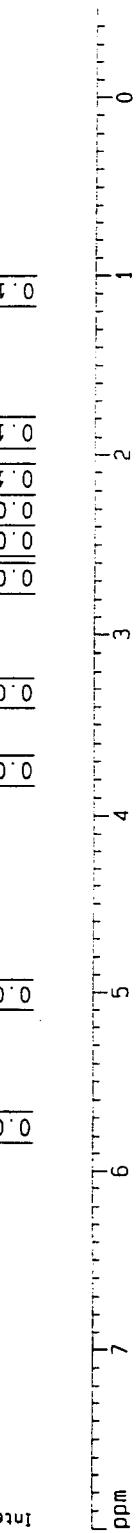
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 INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG 29dc
 TD 65536
 SOLVENT CDCl₃
 NS 33
 DS 0
 SWH 39682.539 Hz
 FIDRES 0.605507 Hz
 AQ 0.8258036 sec
 RG 1024
 DW 12.600 usec
 DE 7.50 usec
 TE 300.0 K
 D1 2.0000000 sec
 d11 0.0300000 sec

===== CHANNEL f1 =====

NUC1 13C
 P1 5.00 usec
 PL1 0.00 dB
 SF01 125.7736214 MHz

===== CHANNEL f2 =====

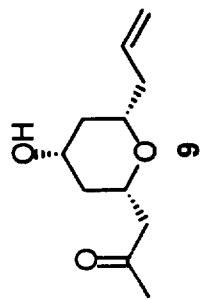
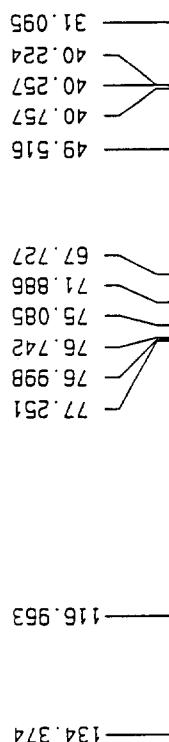
CPDPFG2 13C
 NUC2 1H
 PCP02 100.00 usec
 PL2 120.00 dB
 PL12 20.00 dB
 SF02 500.1320005 MHz

F2 - Processing parameters

S1 32768
 SF 125.7577958 MHz
 WDM EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters

CX 20.00 cm
 F1P 220.000 ppm
 F1 27866.71 Hz
 F2P 0.000 ppm
 F2 0.00 Hz
 PPVCM 11.00000 ppm/cm
 HZCM 1383.33362 Hz/cm

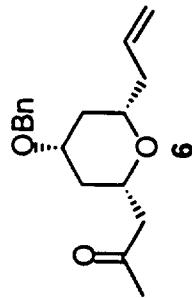


—207.336

ppm

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Current Data Parameters
NAME sk-proton
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters

Date 20001206
Time 17.38
INSTRUM Spect
PROBHD 5 mm Multinu
PULPROG zg
TD 32768
SOLVENT CDCl₃
NS 4
DS 0
SWH 15015.015 Hz
FIDRES 0.458222 Hz
AQ 1.0912244 sec
RG 50.8
DM 33.300 usec
DE 7.50 usec
TE 300.0 K
D1 1.0000000 sec

===== CHANNEL f1 =====

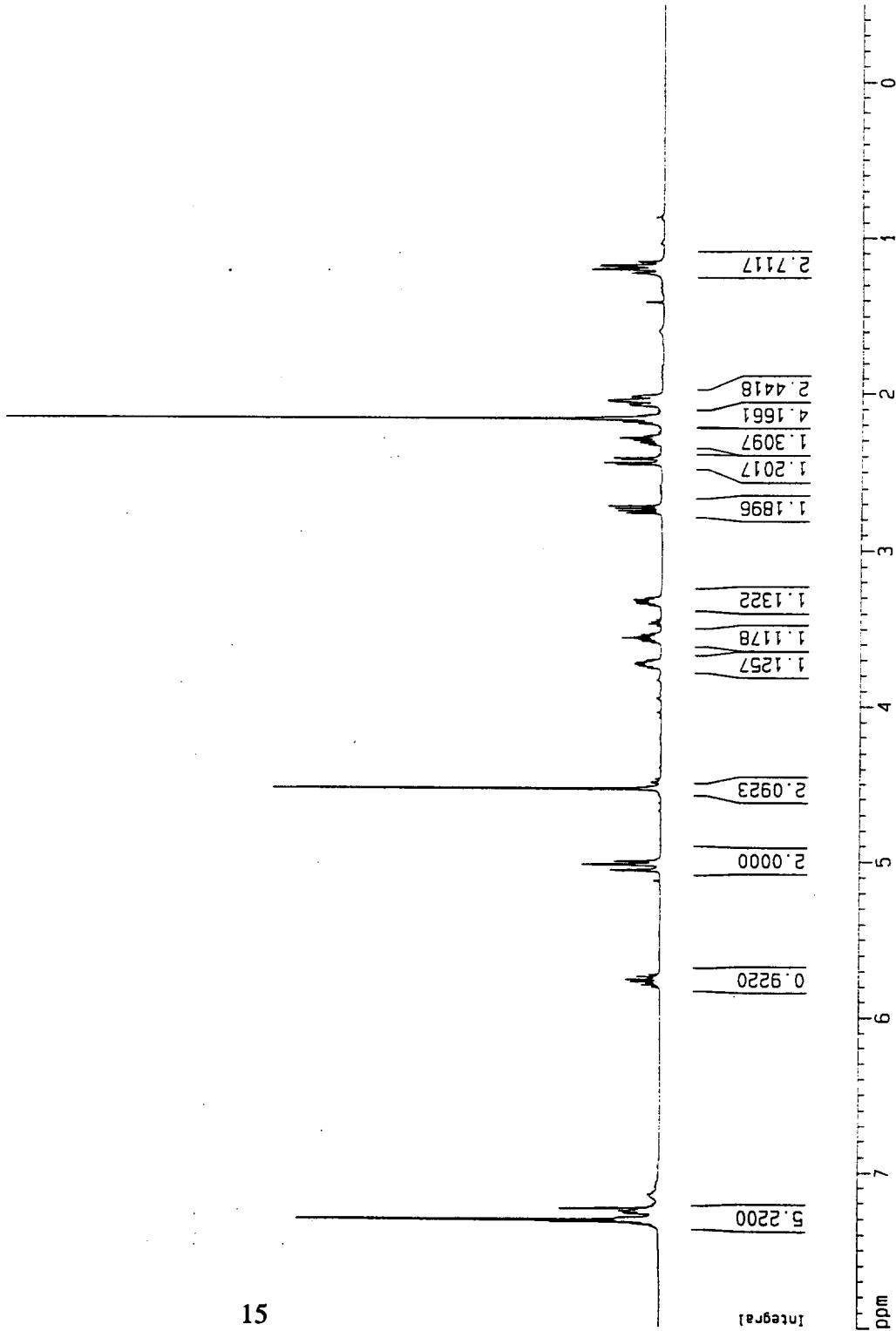
NUC1 1H
P1 5.00 usec
PL1 4.00 dB
SF01 500.1317628 MHz
NDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

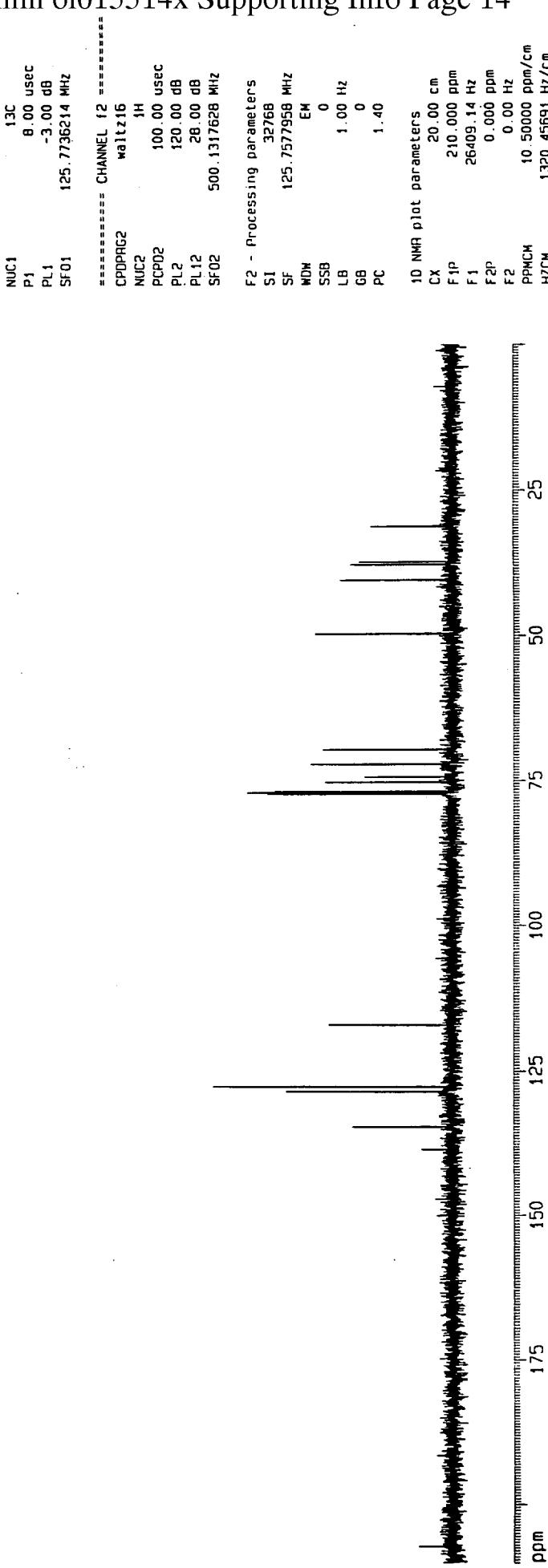
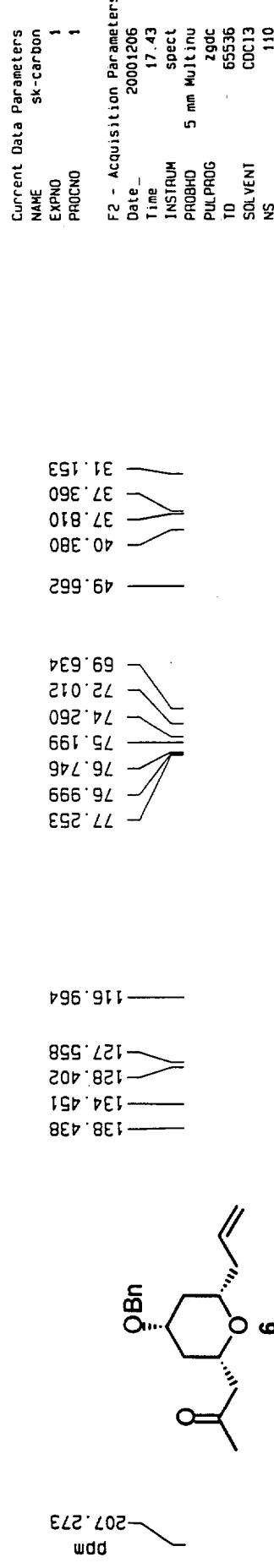
F2 - Processing parameters

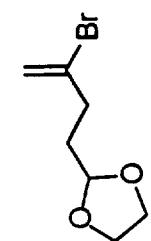
SI 16384
SF 500.1300234 MHz
EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters

CX 20.00 cm
F1P 8.000 ppm
F1 4001.04 Hz
F2P -0.500 ppm
F2 -250.07 Hz
PPMCM 0.42500 ppm/cm
HZCM 212.55537 Hz/cm







Current Data Parameters
NAME dsrh1
EXPNO 1
PROCND 1

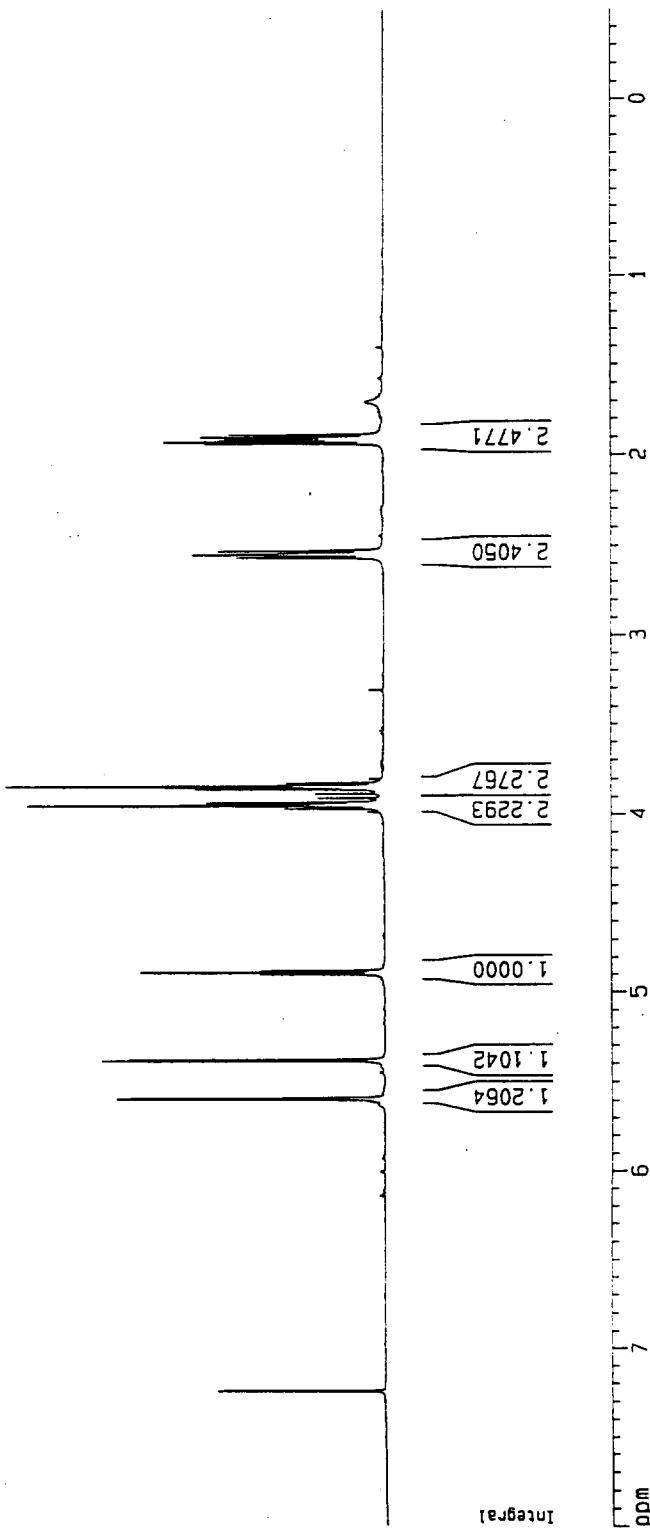
F2 - Acquisition Parameters

Date 20000919
Time 11.46
INSTRUM Spect
PROBHD 5 mm Multinu
PULPROG 32768
TD 32768
SOLVENT CDCl3
NS 8
DS 0
SWH 4888.812 Hz
FIDRES 0.149012 Hz
AQ 3.3554933 sec
RG 80.6
DW 102.400 usec
DE 7.00 usec
TE 300.0 K
D1 1.0000000 sec
P1 7.70 usec
SF01 400.137512 MHz
NUC1 1H
PL1 -6.00 dB

F2 - Processing parameters

CX 20.00 cm
F1P 8.000 ppm
SF 400.1300172 MHz
NDW EN
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

PPMCH 0.42500 ppm/cm
HZCM 170.05525 Hz/cm



Current Data Parameters
 NAME carbon
 EXPNO 1
 PROCN0 1

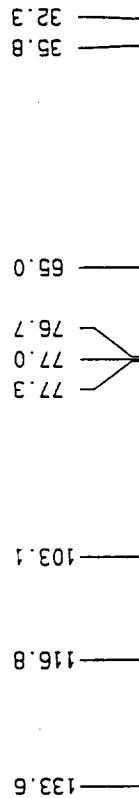
F2 - Acquisition Parameters

Date 20000919
 Time 11:50
 INSTRUM Spect
 PROBHD 5 mm Multinu
 PULPROG zgdc
 TO 65536
 SOLVENT CDCl3
 NS 49
 DS 0
 SWH 29239.766 Hz
 FIDRES 0.446163 Hz
 AQ 1.1207156 sec
 RG 2048
 DW 17.100 usec
 DE 7.50 usec
 TE 300.0 K
 d11 0.03000000 sec
 PL12 24.00 dB
 CPDPRG2 waltz16
 PCP02 105.00 usec
 SF02 400.1315715 MHz
 NUC2 1H
 PL2 0.00 dB
 D1 2.5000000 sec
 P1 8.00 usec
 SF01 100.626545 MHz
 NUC1 13C
 PL1 -6.00 dB

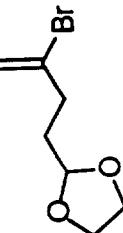
F2 - Processing parameters

SI 32768
 SF 100.6127724 MHz
 MDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.00

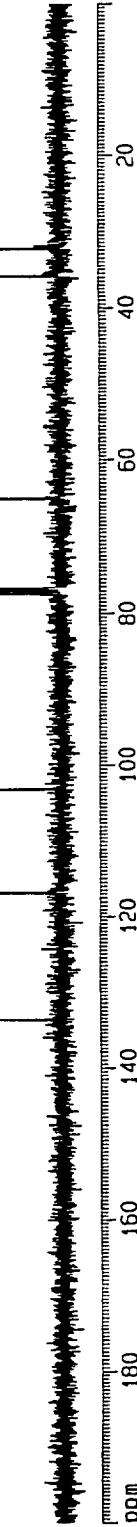
CX 20.00 cm
 F1P 200.00 ppm
 F1 20122.55 Hz
 F2P 0.00 ppm
 F2 0.00 Hz
 PPMCM 10.00000 ppm/cm
 HZCM 1006.12769 Hz/cm



ppm



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Current Data Parameters
 NAME proton
 EXPNO 1
 PROCN0 1

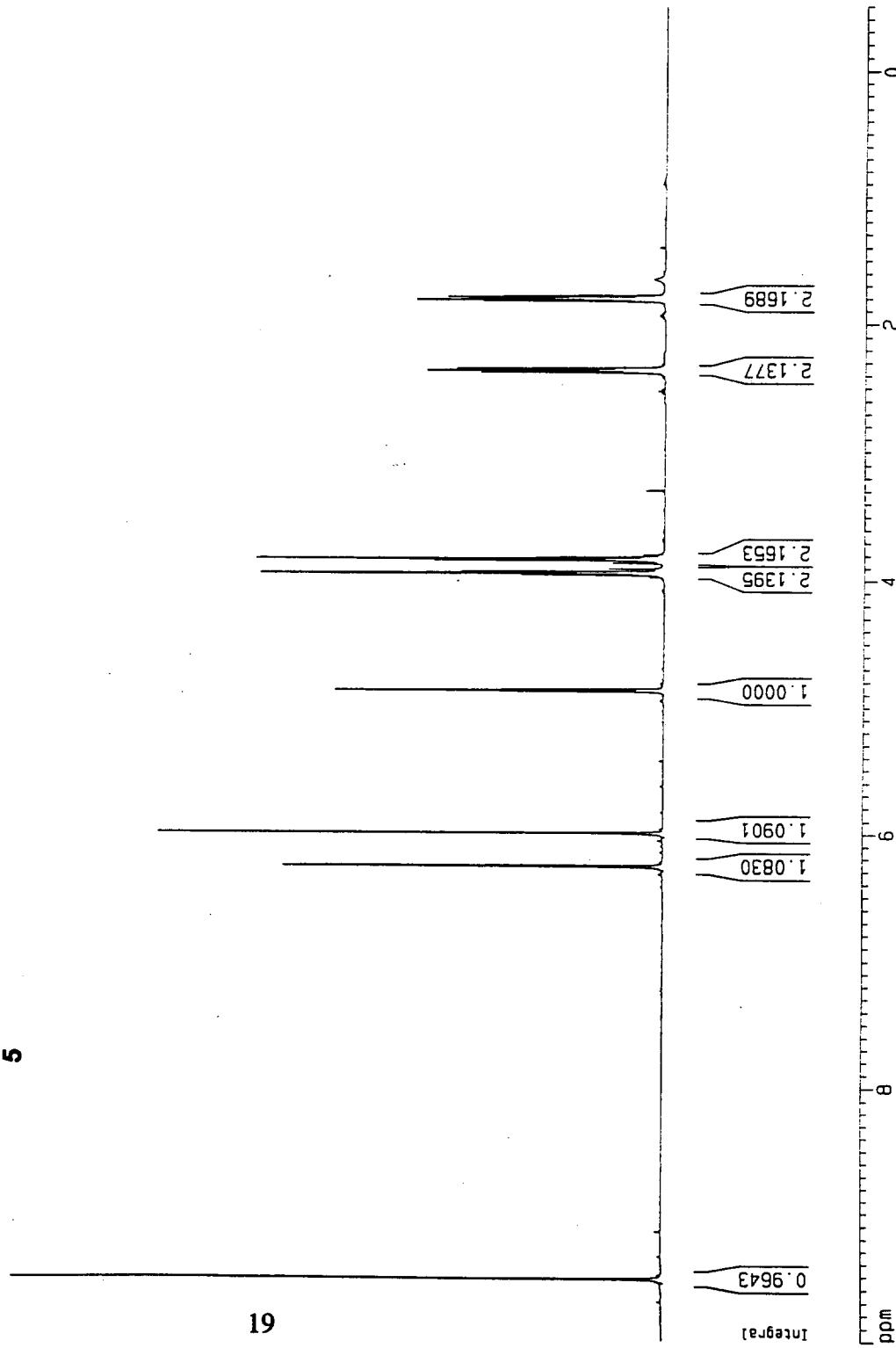
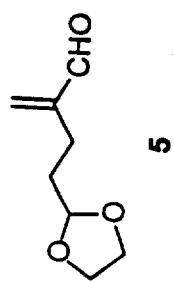
F2 - Acquisition Parameters

Date 20001219
 Time 13.12
 INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG 29
 T0 32768
 SOLVENT CDCl3
 NS 4
 DS 0
 SWH 10000.000 Hz
 FIDRES 0.305176 Hz
 AQ 1.6384500 sec
 RG 128
 DW 50.000 usec
 DE 4.50 usec
 TE 300.0 K
 D1 1.0000000 sec

===== CHANNEL f1 =====

NUC1 1H
 P1 5.00 usec
 PL1 0.00 dB
 SF01 500.1338178 MHz
 MDW 0
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

F2 - Processing parameters
 SI 16384
 SF 500.1300237 MHz
 EM 0
 DW 20.00 ppm
 F1P 10.000 ppm
 F1 5001.30 Hz
 F2P -0.500 ppm
 F2 -250.07 Hz
 PPICM 0.52500 ppm/cm
 HZCM 262.56827 Hz/cm



Current Data Parameters
 NAME carbon 1
 EXPNO 1
 PROCN0 1

F2 - Acquisition Parameters

Date 20001219
 T1me 13.15
 INSTRUM spect
 PROBID 5 mm QNP 1H
 PULPROG 29dc
 TD 65536
 SOLVENT Toluol
 NS 55
 DS 0
 SWH 39682.539 Hz
 FIDRES 0.605507 Hz
 AQ 0.8258036 sec
 AG 1024
 DW 12.600 usec
 DE 7.50 usec
 TE 300.0 K
 D1 2.0000000 sec
 D11 0.03000000 sec

===== CHANNEL f1 =====

NUC1 13C
 P1 5.00 usec
 PL1 0.00 dB
 SF01 125.7736214 MHz

===== CHANNEL f2 =====

CPDOPG2 13C
 NUC2 1H
 PCP02 100.00 usec
 PL2 120.00 dB
 PL12 20.00 dB
 SF02 500.1320005 MHz

F2 - Processing parameters

S1 32768
 SF 125.7577936 MHz
 MW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

10 NMR plot parameters

CX 20.00 cm
 F1P 200.00 ppm
 F1 25151.56 Hz
 F2P 0.0000 ppm
 F2 0.00 Hz
 PPMM 10.00000 ppm/cm
 HZCM 1257.573600 Hz/cm

22.321
 31.731

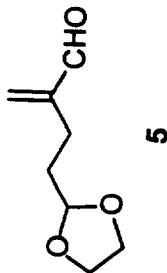
64.923
 76.748
 77.000
 77.255

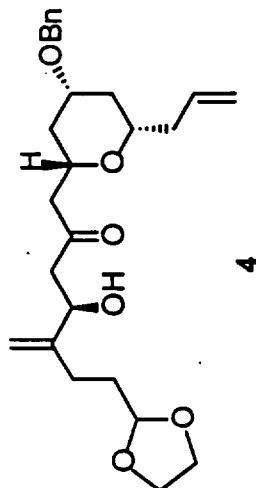
103.750

134.046

149.512

194.380
 ppm





Current Data Parameters
 NAME proton
 EXPNO 1
 PROCN0 1

F2 - Acquisition Parameters

Date 20001220
 Time 20.17
 INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG 29
 TD 32768
 SOLVENT CDCl₃
 NS 4
 DS 0
 SWH 10000.000 Hz
 FIDRES 0.305176 Hz
 AQ 1.6384500 sec
 AG 256
 DM 50.000 usec
 DE 4.50 usec
 TE 300.0 K
 D1 1.00000000 sec

===== CHANNEL f1 =====

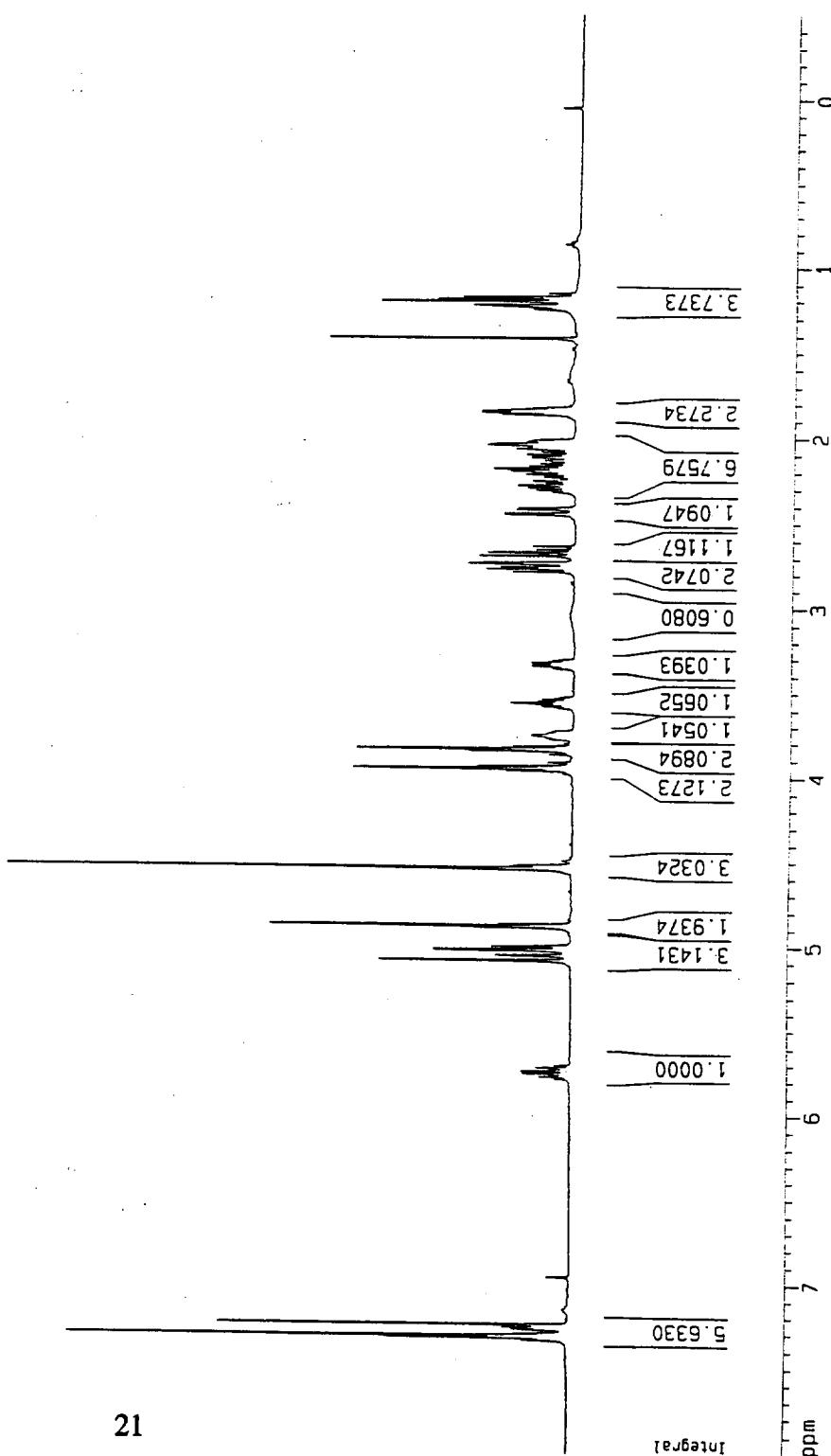
NUC1 1H
 P1 5.00 usec
 PL1 0.00 dB
 SF01 500.1300231 MHz

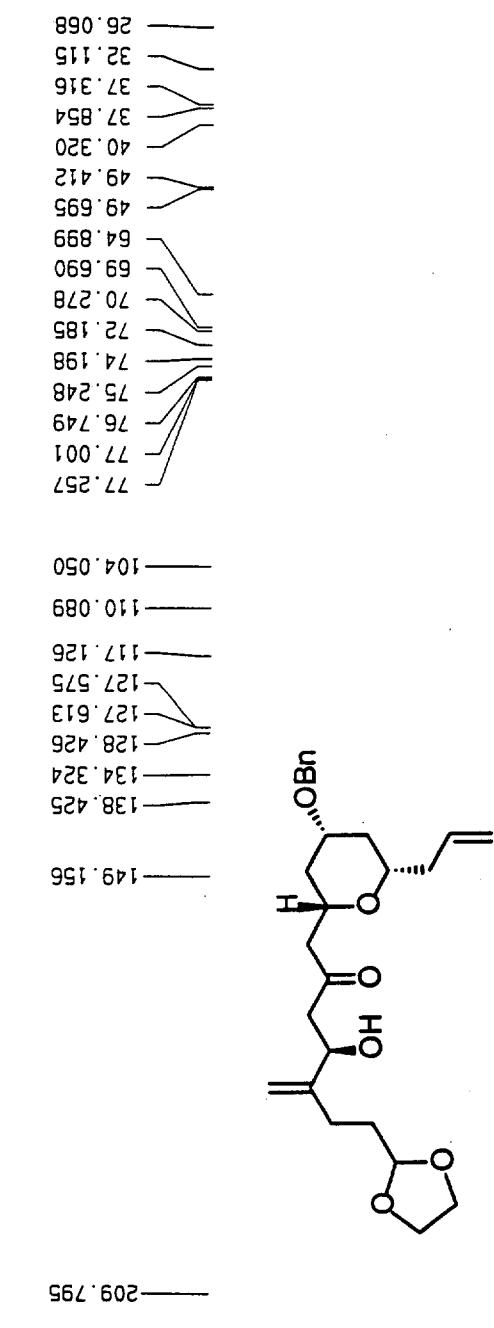
F2 - Processing parameters

S1 16384
 SF 500.1300231 MHz
 MDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters

CX 20.00 cm
 F1P 8.000 ppm
 F1 400.104 Hz
 F2P -0.500 ppm
 F2 -250.07 Hz
 PPMCM 0.42500 ppm/cm
 HZCM 212.55527 Hz/cm





Current Data Parameters

NAME	carbon
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date _	20001221
Time _	0.37
INSTRUM	spect
PROBHD	5 mm QNP 1H
PULPROG	zgdc
TD	65536
SOLVENT	C02C12
NS	1702
DS	0
SWH	39682.539 Hz
FDRES	0.60507 Hz
AQ	0.8258036 sec
RG	1024
DW	12.600 usec
DE	7.50 usec
TE	300.0 K
D1	2.0000000 sec
d11	0.0300000 sec

===== CHANNEL f1 =====

NUC1	13C
P1	5.00 usec
PL1	0.00 dB
SF01	125.7736214 MHz

===== CHANNEL f2 =====

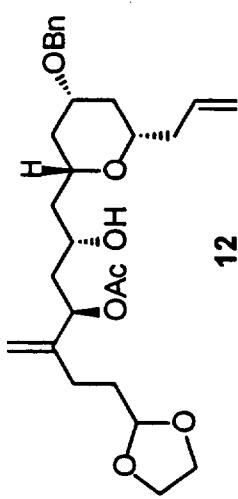
CPDPFG2	Waltz16
NUC2	1H
PCPD2	100.00 usec
PL2	120.00 dB
PL12	20.00 dB
SF02	500.1320005 MHz

F2 - Processing parameters

SI	32768
SF	125.7577932 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

10 NMR plot parameters

CX	20.00 cm
F1P	220.000 ppm
F1	27656.71 Hz
F2P	0.000 ppm
F2	0.00 Hz
PPMCH	11.00000 ppm/cm
HZCM	1383.33569 Hz/cm



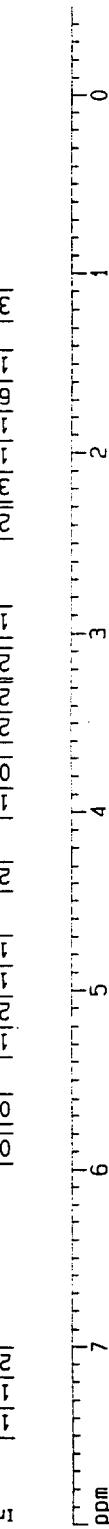
Current Data Parameters
NAME proton 1
EXPNO 1
PROCNO 1

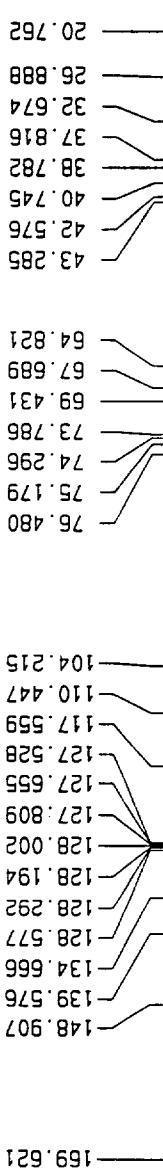
F2 - Acquisition Parameters
Date 20001221
Time 16:01
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg32
TD 32768
SOLVENT C6D6
NS 4
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 1.6384500 sec
RG 32
DW 50.000 usec
DE 4.50 usec
TE 300.0 K
D1 1.00000000 sec

==== CHANNEL f1 ======
NUC1 1H
P1 5.00 usec
PL1 0.00 dB
SF01 500.1318178 MHz
NDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

F2 - Processing parameters
SI 16384
SF 500.1300620 MHz
NDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

10 NMR plot parameters
CX 20.00 cm
F1P 8.000 ppm
F1 4001.04 Hz
F2P -0.500 ppm
F2 -250.07 Hz
PPCM 0.42500 ppm/cm
HZCM 212.55528 Hz/cm





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Current Data Parameters

NAME	carbon
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date	20001221
Time	18.07
INSTRUM	spec
PROBHD	5 mm QNP
PULPROG	zdc
TD	65536
SOLVENT	CDCl ₃
NS	101
D1	0
SWH	39682.539 Hz
FLDRES	0.605507 Hz
AQ	0.8258036 sec
RG	1024
DW	12.600 usec
DE	7.50 usec
TE	300.0 K
D1	2.0000000 sec
d11	0.0300000 sec

==== CHANNEL 11 =====

CPDPFG2	13C
NUC1	waltz16
P1	5.00 usec
PL1	0.00 dB
SF01	125.7736214 MHz

==== CHANNEL 12 =====

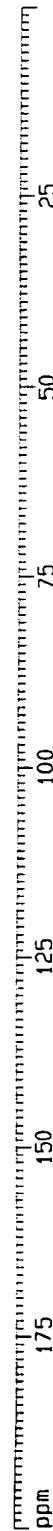
CPDPFG2	13C
NUC1	waltz16
P1	100.00 usec
PL1	120.00 dB
SF01	500.1320005 MHz

F2 - Processing parameters

S1	32768
SF	125.757766 MHz
MDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	20.00 ppm
F1P	200.00 ppm
F1	25151.55 Hz
F2P	0.00 ppm
F2	0.00 Hz
PPMOM	10.00000 ppm/cm
HZCM	1257.57776 Hz/cm



Current Data Parameters
 NAME proton
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters

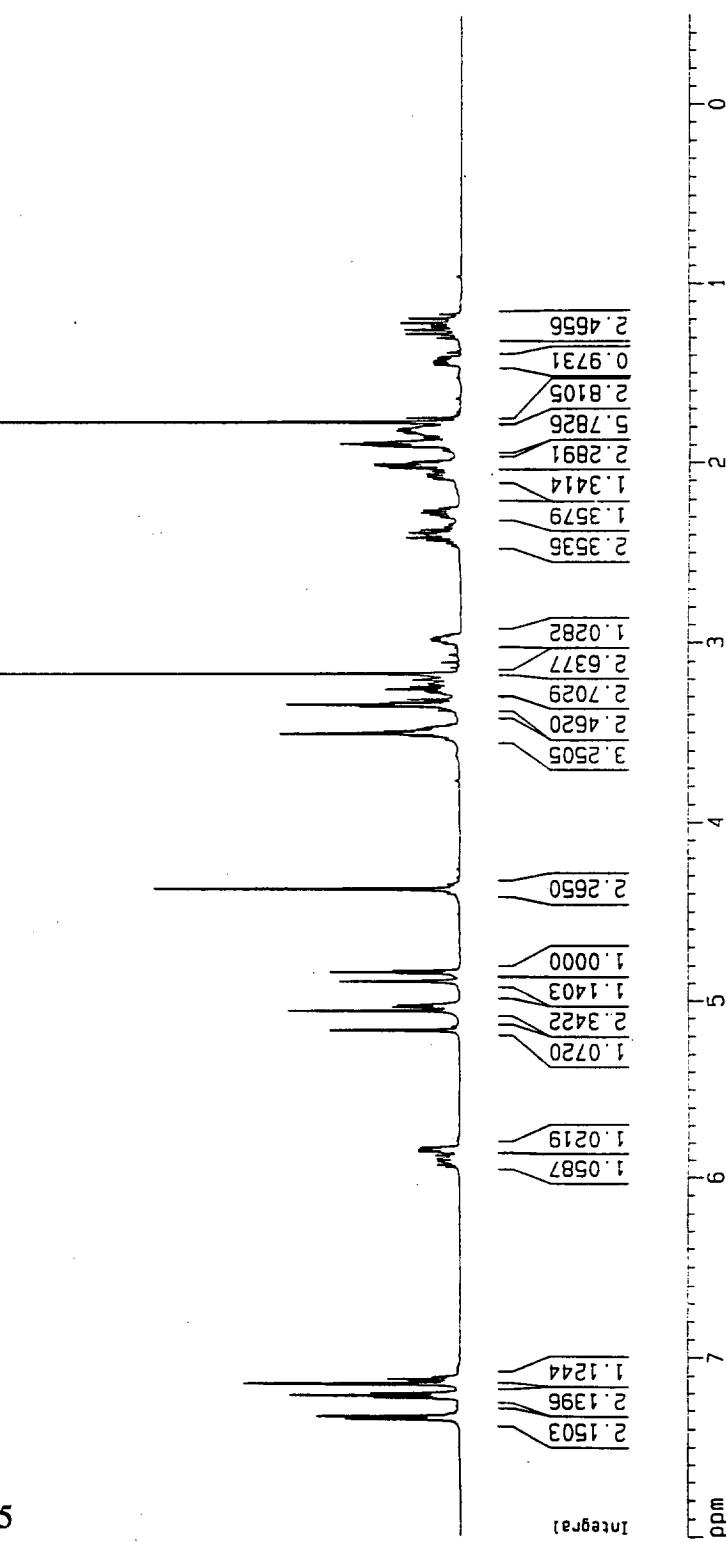
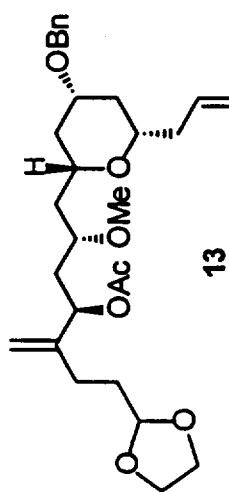
Date 20001222
 Time 10.24
 INSTRUM Spect
 PROBHD 5 mm QNP 1H
 PULPROG zg
 TD 32768
 SOLVENT C6D6
 NS 4
 DS 0
 SWH 10000.000 Hz
 FIDRES 0.305176 Hz
 AQ 1.6384500 sec
 RG 32
 DW 50.000 usec
 DE 4.50 usec
 TE 300.0 K
 D1 1.0000000 sec

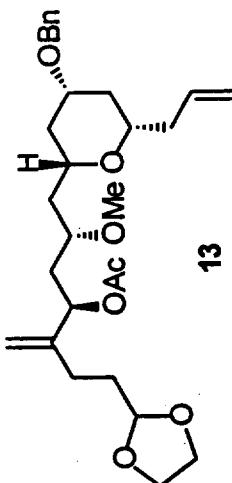
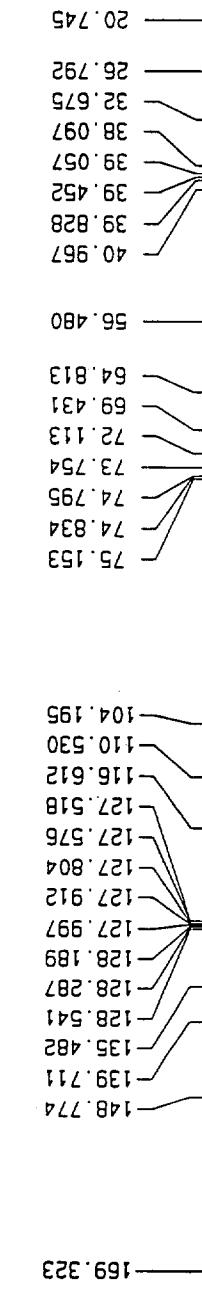
===== CHANNEL f1 =====

NUC1 1H
 P1 5.00 usec
 PL1 0.00 dB
 SF01 500.1318178 MHz
 MW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

F2 - Processing parameters

SI 16384
 SF 500.1300526 MHz
 MW EM
 F2P -0.500 ppm
 F2 -250.07 Hz
 PPMCM 0.42300 ppm/cm
 HZCM 212.55528 Hz/cm





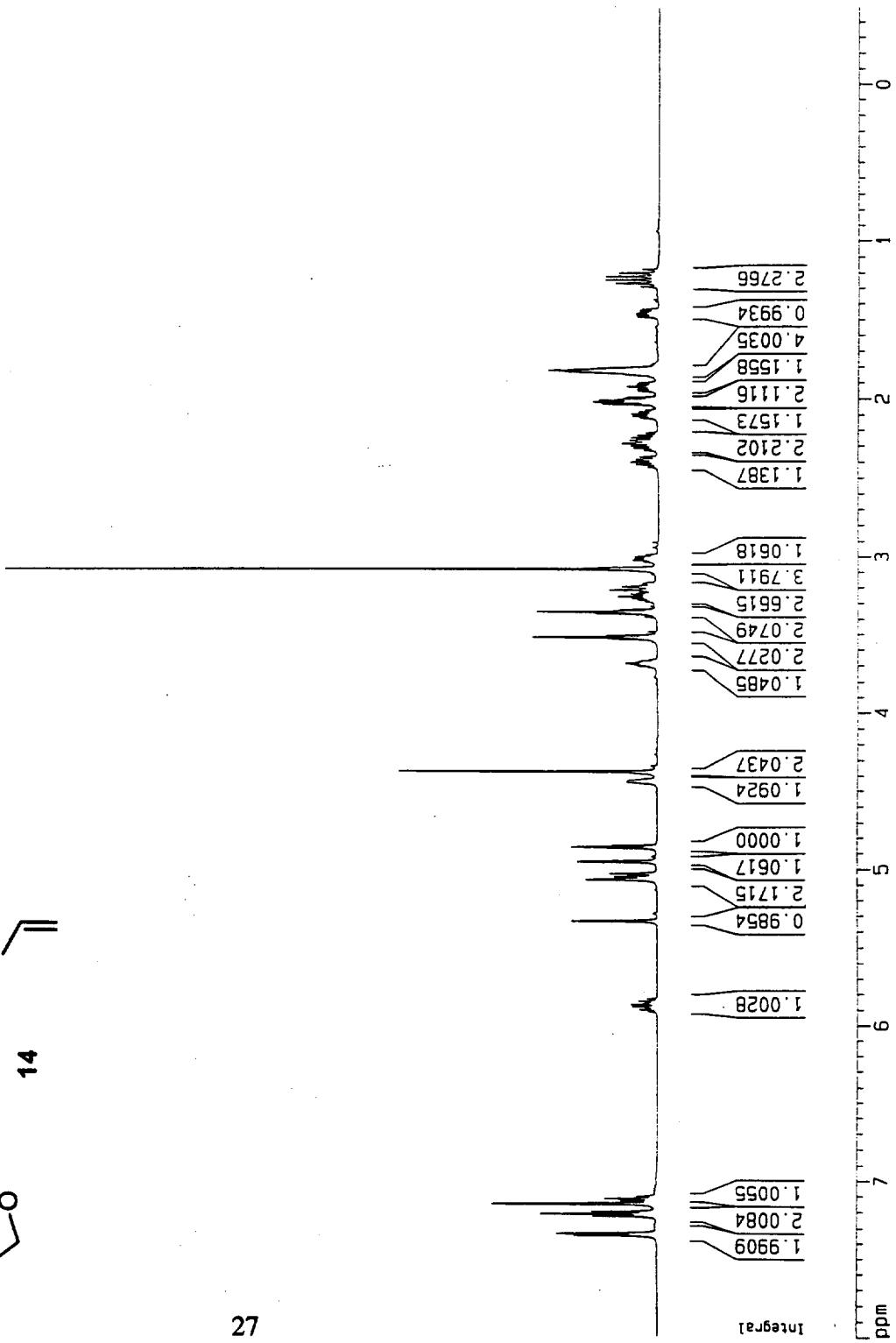
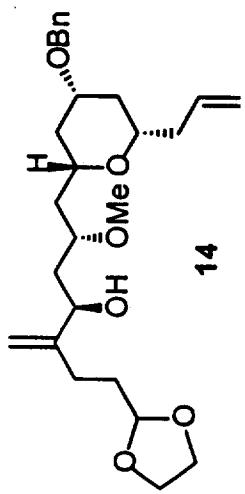
Current Data Parameters
NAME proton EXPNO 1 PROCNO 1

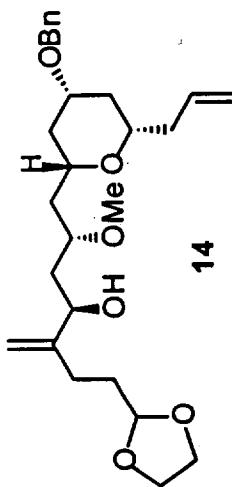
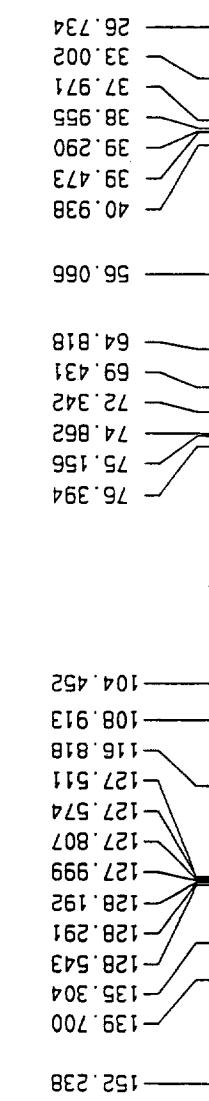
F2 - Acquisition Parameters
 Date _ 20001222
 Time 14.40
 INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG zg
 T0 32768
 C606
 SOLVENT 4
 NS 0
 DS 0
 SWH 10000.000 Hz
 FIDRES 0.305176 Hz
 AQ 1.6384500 sec
 RG 32
 DW 50.000 usec
 DE 4.50 uK
 TE 300.0 K
 D1 1.00000000 sec

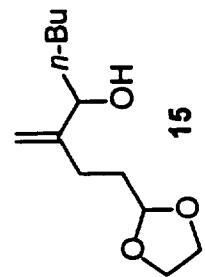
```
===== CHANNEL f1 =====
NUC1          1H
P1            5.00 usec
PL1           0.00 dB
SF01         500.1318178 MHz
```

F2 - Processing parameters	
SI	16384
SF	500.1300626 MHz
MOW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

1D NMR plot parameters	
CX	20.00 cm
F1P	8.000 ppm
F1	4001.04 Hz
F2P	-0.500 ppm
F2	-250.07 Hz
PPMCH	0.42500 ppm/cm
H7CM	212.55528 Hz/cm





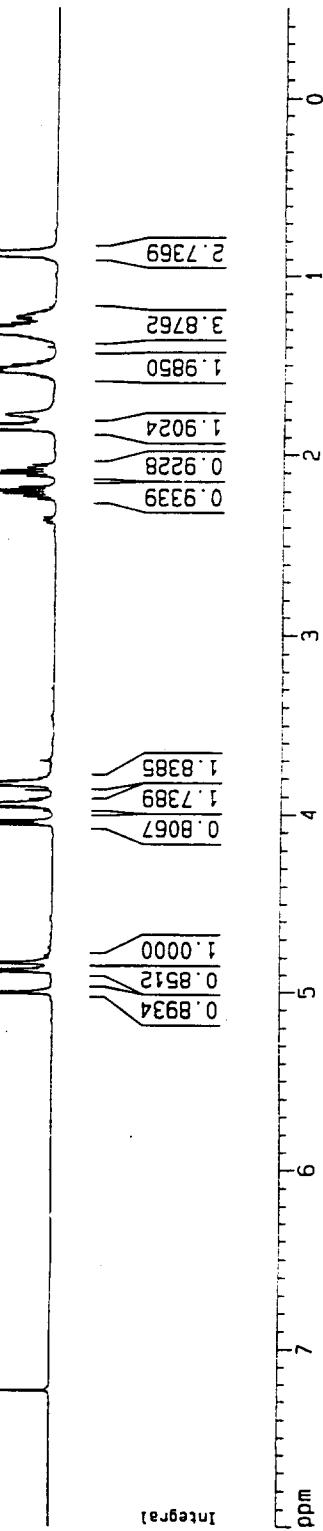


Current Data Parameters
 NAME sk-proton
 EXPNO 3
 PROCNO 1

F2 - Acquisition Parameters
 Date 2000/12/06
 Time 17:52
 INSTRUM spect
 PROBHD 5 mm Multinu
 PULPROG zg32768
 T0 32768
 SOLVENT CDCl₃
 NS 4
 DS 0
 SWH 15015.015 Hz
 FIDRES 0.453222 Hz
 AQ 1.0912244 sec
 RG 40.3
 DW 33.300 usec
 DE 7.50 usec
 TE 300.0 K
 D1 1.0000000 sec

===== CHANNEL f1 =====:
 NUC1 1H
 P1 5.00 usec
 PL1 4.00 dB
 SF01 500.1317628 MHz

F2 - Processing parameters
 S1 16384
 SF 500.1300234 MHz
 NDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



Current Data Parameters
 NAME sk-carbon
 EXPNO 1
 PROCN0 1

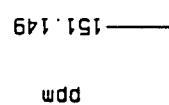
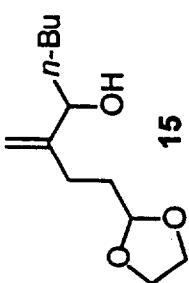
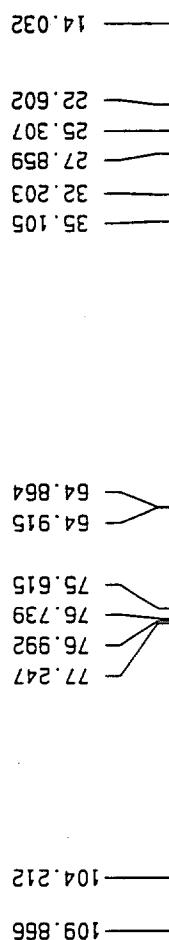
F2 - Acquisition Parameters
 Date 20001206
 Time 17:56
 INSTRUM spect
 PROBHD 5 mm Multinu
 PULPROG 20dc
 TD 65536
 SOLVENT CDCl3
 NS 54
 DS 1
 SWH 39602.939 Hz
 FIDRES 0.605017 Hz
 AQ 0.8258036 sec
 RG 9195.2
 DW 12.600 usec
 DE 4.50 usec
 TE 300.0 K
 D1 2.0000000 sec
 d11 0.0300000 sec

===== CHANNEL f1 =====
 CPDPFG2 NUC1 13C
 P1 8.00 usec
 PL1 -3.00 dB
 SF01 125.7736214 MHz

===== CHANNEL f2 =====
 CPDPFG2 NUC2 1H
 PCPD02 100.00 usec
 PL2 120.00 dB
 PL12 28.00 dB
 SF02 500.1317628 MHz

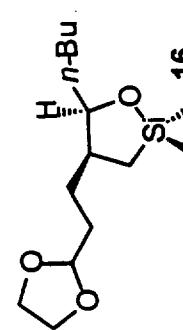
F2 - Processing parameters
 SI 32768
 SF 125.7577958 MHz
 MDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC. 1.40

10 NMR pilot parameters
 CX 20.00 cm
 F1P 160.000 ppm
 F1 20121.25 Hz
 F2P 0.000 ppm
 F2 0.00 Hz
 PPRCM 8.00000 ppm/cm
 HZCM 1006.06238 Hz/cm

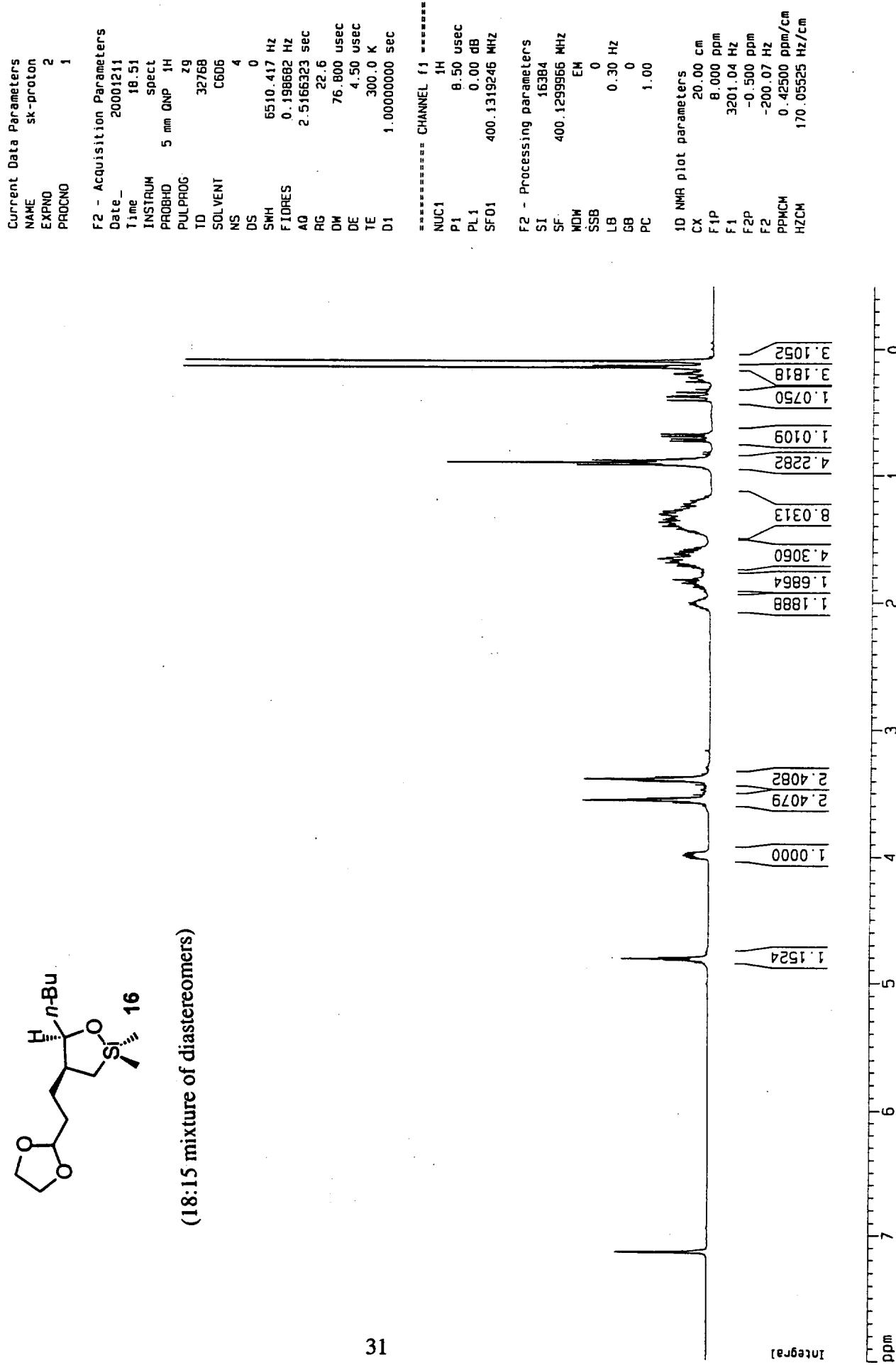


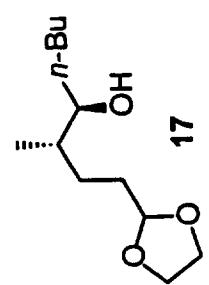
ppm

30

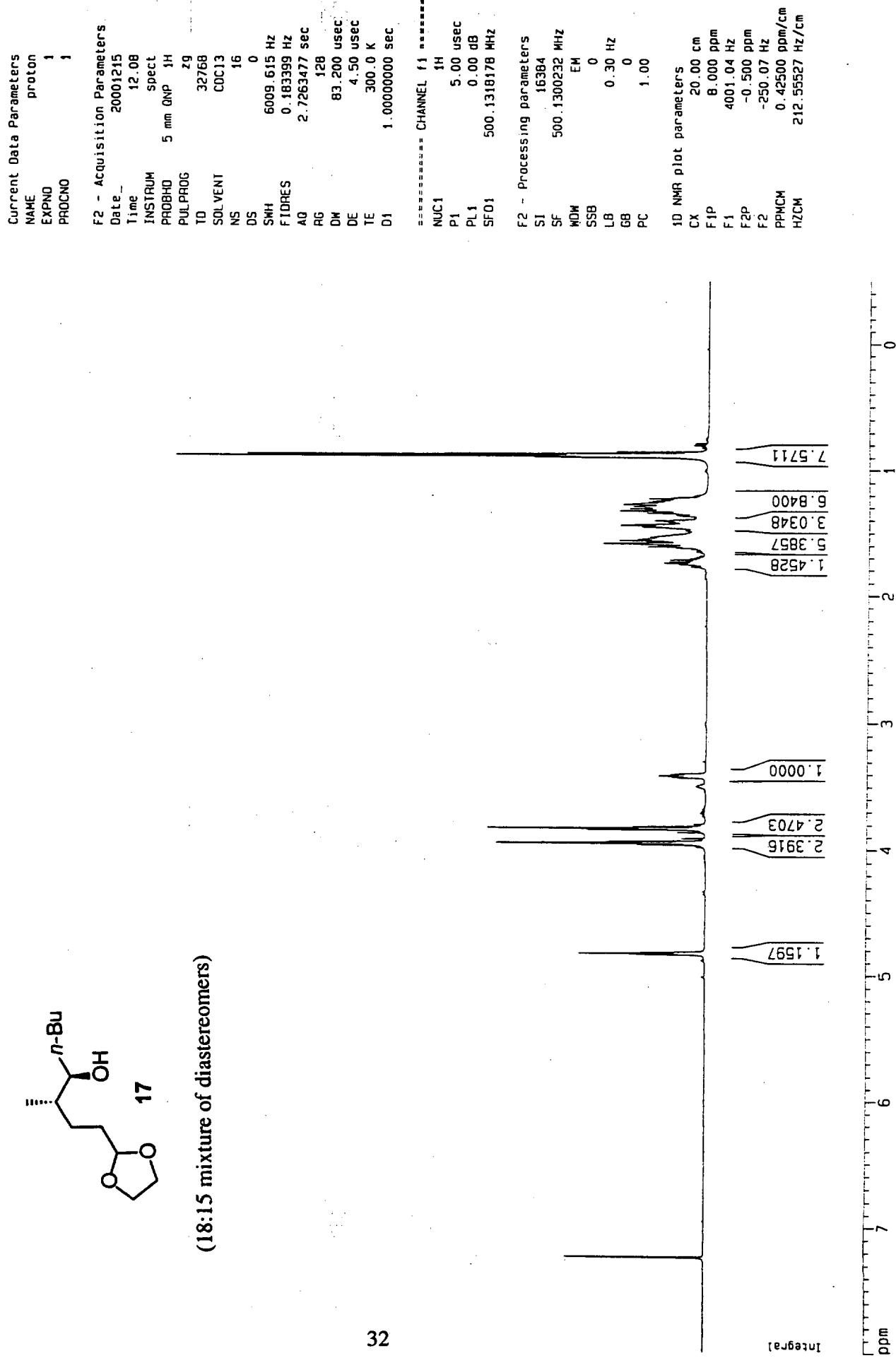


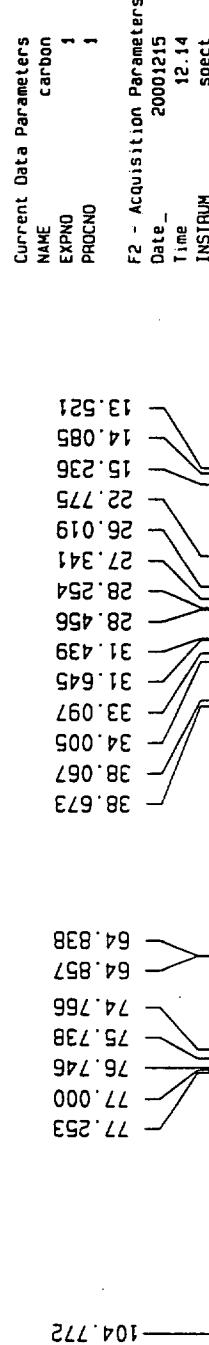
(18:15 mixture of diastereomers)





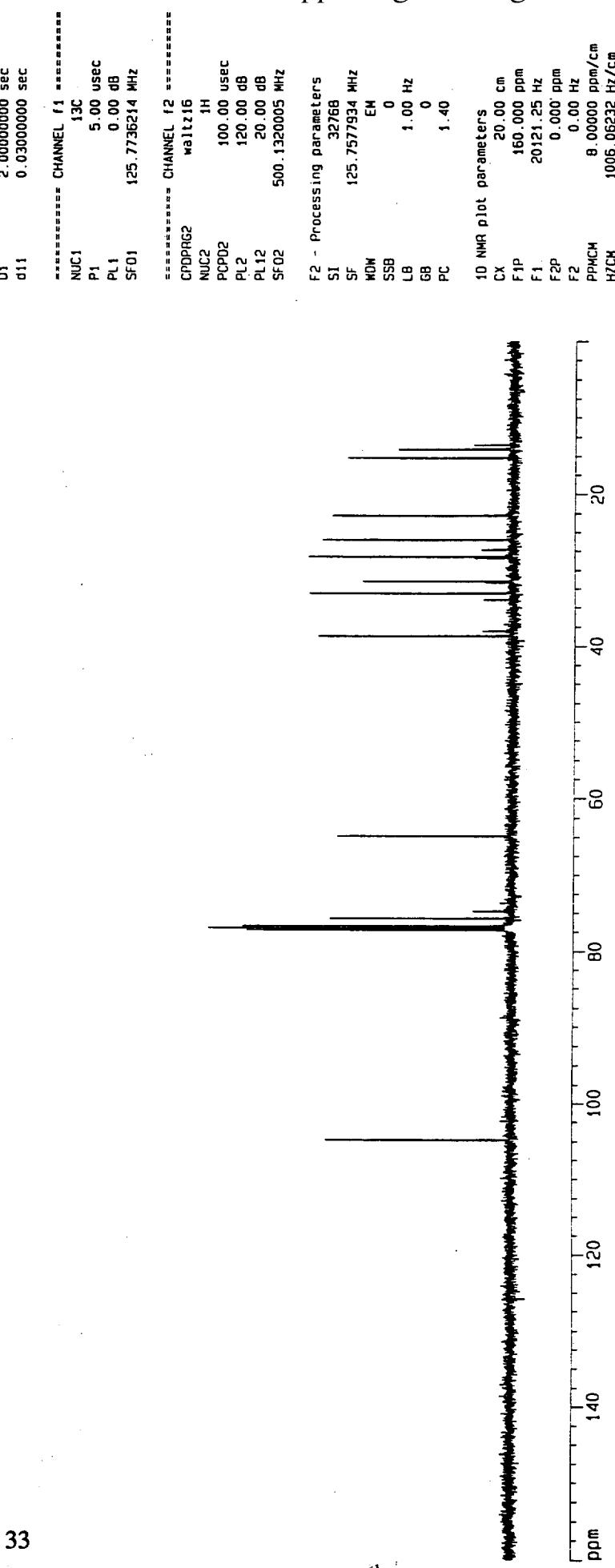
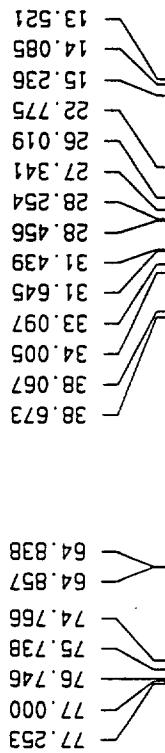
(18:15 mixture of diastereomers)

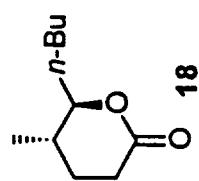




ppm

104.772





Current Data Parameters
 NAME sk-proton
 EXPNO 1
 PROCN0 1

F2 - Acquisition Parameters

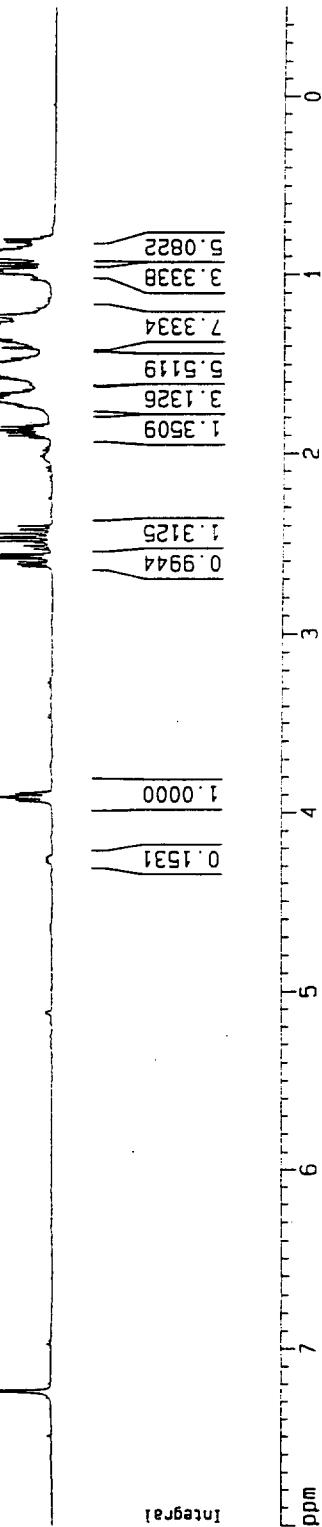
Date _	20001218
Time	18.17
INSTRUM	spect
PROBID	5 mm QNP 1H
PULPROG	2.9
TD	32768
SOLVENT	CDC13
NS	16
DS	0
SWH	650.417 Hz
TDRES	0.198682 Hz
AQ	2.5066323 sec
RG	322.5
DW	76.800 usec
DE	4.50 usec
TE	300.0 K
01	1.0000000 sec

===== CHANNEL f1 =====

NUC1	1H
P1	8.50 usec
PL1	0.00 dB
SF01	400.1319246 MHz
NDW	0
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

F2 - Processing parameters

CX	20.00 cm
F1P	8.000 ppm
F1	3201.04 Hz
F2P	-0.500 ppm
F2	-200.07 Hz
PPMCM	0.42850 ppm/cm
HZCM	170.055325 Hz/cm



Current Data Parameters
NAME carbon 1
EXPO 1
PROCNO 1

F2 - Acquisition Parameters

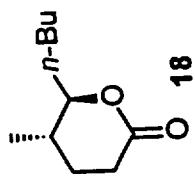
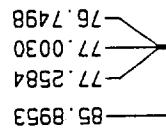
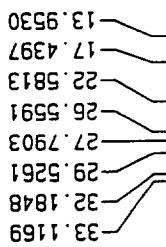
Date 20001218
Time 16.32
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgdc
TD 65536
SOLVENT T01
NS 418
DS 0
SWH 39682.539 Hz
FIDRES 0.605507 Hz
AQ 0.828036 sec
RG 1024
DW 12.600 usec
DE 7.50 usec
TE 300.0 K
D1 2.0000000 SEC
D11 0.0300000 SEC

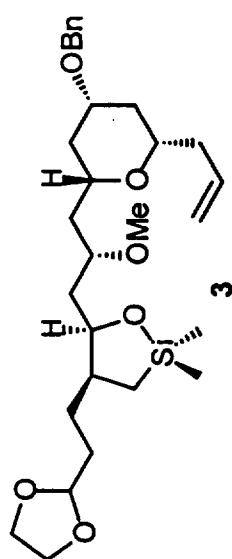
***** CHANNEL f1 *****
NUC1 13C
P1 5.00 usec
PL1 125.7736214 MHz
SF01 500.1320005 MHz

***** CHANNEL f2 *****
CPDPFG2 Wattz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 20.00 dB
SF02 500.1320005 MHz

F2 - Processing parameters
SI 32768
SF 125.757922 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR pilot parameters
CX 20.00 cm
F1P 200.000 ppm
F1 25151.56 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCH 10.00000 ppm/cm
H2CM 1257.57768 Hz/cm





Current Data Parameters
NAME proton
EXPN0 1
PROCNO 1

F2 - Acquisition Parameters

Date 20001222
Time 17.25
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG 32768
TD 32768
SOLVENT C6D6
NS 4
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 1.6384500 sec
RG 32
DW 50.000 usec
DE 4.50 usec
TE 300.0 K
D1 1.0000000 sec

F2 - CHANNEL f1

NUC1 1H
SI 5.00 usec
P1 0.00 dB
PL1 500.1318178 MHz
SF01 500.1300620 MHz

F2 - Processing parameters

SI 16384
SF 500.1300620 MHz
MDW 0
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters

CX 20.00 cm
F1P 8.000 ppm
F1 4001.04 Hz
F2P -0.500 ppm
F2 -250.07 Hz
PPMCM 0.42500 ppm/cm
HZCM 212.55528 Hz/cm

